UNIVERSITI TEKNOLOGI MARA

EPOXIDATION OF PALM OIL USING SYNTHESIZED TITANIUM-SILICA CATALYST AND INVESTIGATION ON THE PROPERTIES OF ACRYLATED EPOXIDIZED PALM OIL

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

Catalytic palm oil epoxidation using titanium-grafted silica, hydrogen peroxide, and peroxoformic acid was carried out at 60°C in a fixed batch reactor. Titanium-grafted silica with different percentages of silica content was prepared through sol-gel hydrolysis and was utilized in epoxidation of palm oil. They were characterized by X-Ray Diffractometer (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscope (FESEM) and Nuclear Magnetic Resonance (^{1}H-NMR). The Ti–O–Si bonds were detected at 960 cm^{-1} in Ti-Si 0.5 and exhibited highest yield of epoxidized palm oil (EPO) in the epoxidation process which is 86% conversion of unsaturation in palm oil to epoxy groups. New peaks observed in the range of δ 2.4 ppm to δ 3.6 ppm in the ^{1}H-NMR spectrum of EPO belong to protons of the epoxy cyclic ring group, CH-O-CH confirming successful epoxidation of palm oil using the prepared catalyst. For the catalyst recycle study, Ti-Si 0.5 withstood up to twice catalytic run with negligible changes in their selectivity and with a loss in their specific activity. This study indicated the true nature of heterogeneous catalysis on Ti-Si 0.5. An acrylated epoxidized palm oil (AEPO) was synthesized from EPO through ring opening of the oxirane group using acrylic acid as the ring opening agent. The %OOC reached 0.88% with acid value was 48.25. The derivatised of acrylic acid of AEPO was detected at δ 5.8 ppm to δ 6.5 ppm indicated the appearance of acrylate $C\underline{H}=CH_2$. The new bands which may attributed to the vinyl functionality 1624 cm^{-1}, $CH_2=CH\text{-}COO$ and also at absorption band of 961cm^{-1}, =C-H as can be seen in FTIR result. The increased in viscosity of AEPO, 497.5 Cps was due to the -OH groups that was confirmed by appearance of peak at 3417cm^{-1} in FTIR. The synthesized AEPO was mixed with four different composition of photoinitiator and cured under ultra-violet radiation. the preparation of AEPO films in UV curing adhesive, AEPO 2wt% have the highest surface hardness. The AEPO 2wt% has the highest gel content whereas the AEPO 2.5wt% had the lowest gel content. The increasing of photoinitiator from 1wt% to 2.5wt% in AEPO samples except for AEPO 1.5wt% showed increasing in adhesion strength.

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CHAPTER ONE
INTRODUCTION

1.1 BACKGROUND OF STUDY

Chemical modifications of vegetable oils are significant in developing industrial products based on renewable feed-stocks (Hu, Willey, & Notari, 2003). Vegetable oils also known as fats or lipids are composed primarily of triesters of glycerol with fatty acids and are generally called triglycerides (Abdullah & Salimon, 2010). In particular, oils and fats of vegetable and animal origin exhibited the largest fraction of current consumption of renewable raw materials in the chemical industry, providing applications that cannot be met by petrochemicals (Lligadas, Ronda, Galia, Biermann, & Metzer, 2005).

Unsaturated fat were the component of interest in epoxidation of vegetable oils due to the presence of olefinic bonds required in the reaction. Epoxidation was carried on by the reaction of peroxyacids (peracids) and olefinic double bonds to form an oxirane which is epoxy group (Tellez, Santiago, & Lopez, 2009). Soybean oil which has 84.5% of unsaturated fats allowed successful production of epoxidized soybean oil industrially. This is due to the high degree of unsaturated fats present in soybean oil. Epoxies are valuable commercial products. Specifically, epoxidized triglycerides have been used as diluents, lubricants, coatings and stabilizers in polyvinyl chloride (Meshram, Puri, & Patil, 2011). They are known to be non-volatile, phthalate-free, extraction and migration resistant plasticizers (Tellez, et al., 2009); and have been prepared from linseed, rapeseed, olive, corn, and sunflower oils.

Palm oil contains triglycerides which open up the possibilities for chemical modifications such as acrylation, epoxidation, carboxylation, isomerization, hydroxylation, oxidative cleavage, and hydrogenation (Cativiela, Fraile, Garcia, & Mayoral, 1996). These reactions are mainly recognized by severe reaction conditions, strong reactants, and suitable catalysts. Palm oil consists of 56.6% of unsaturated fats (oleic and linoleic acids), and 43.4% of saturated fats (palmitic acid) (Hu, et al., 2003).